CEMENT AND LIME MANUFACTURE

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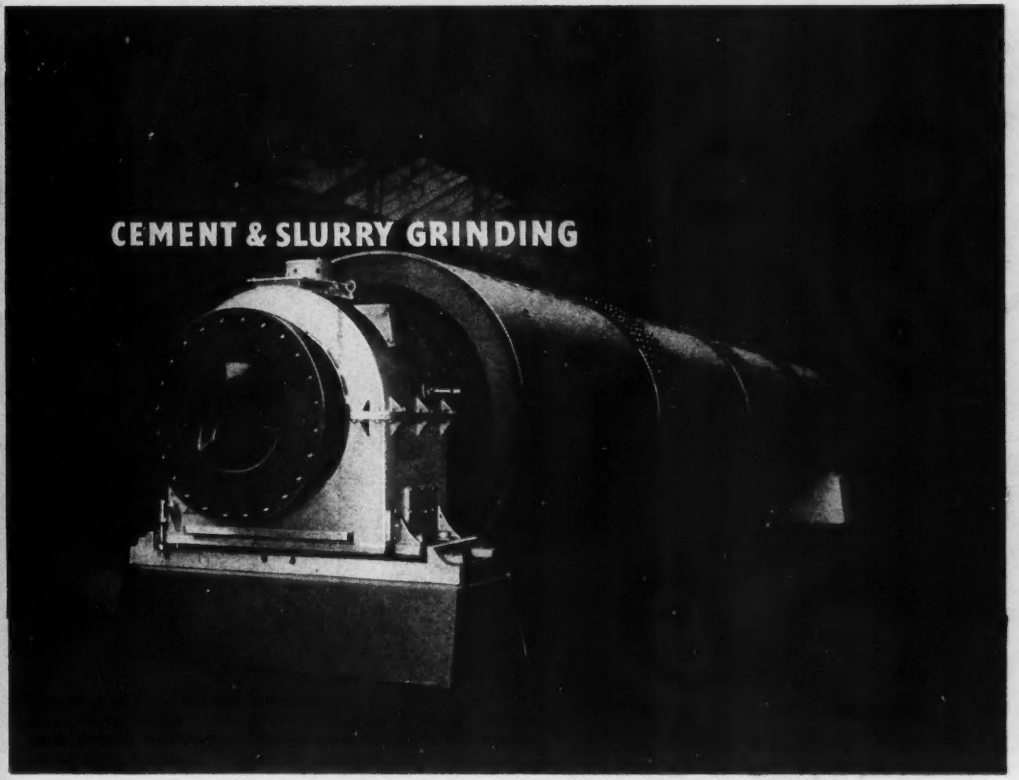

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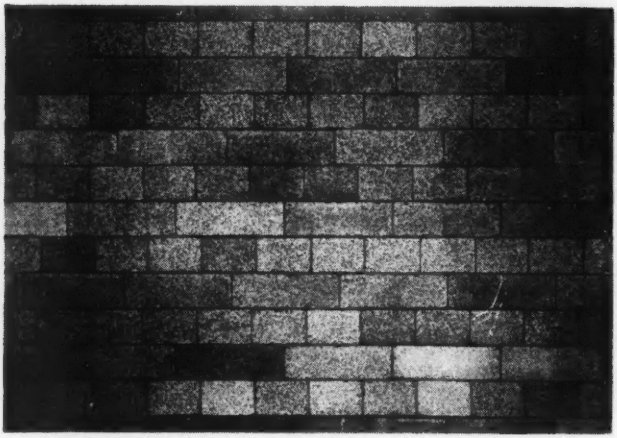

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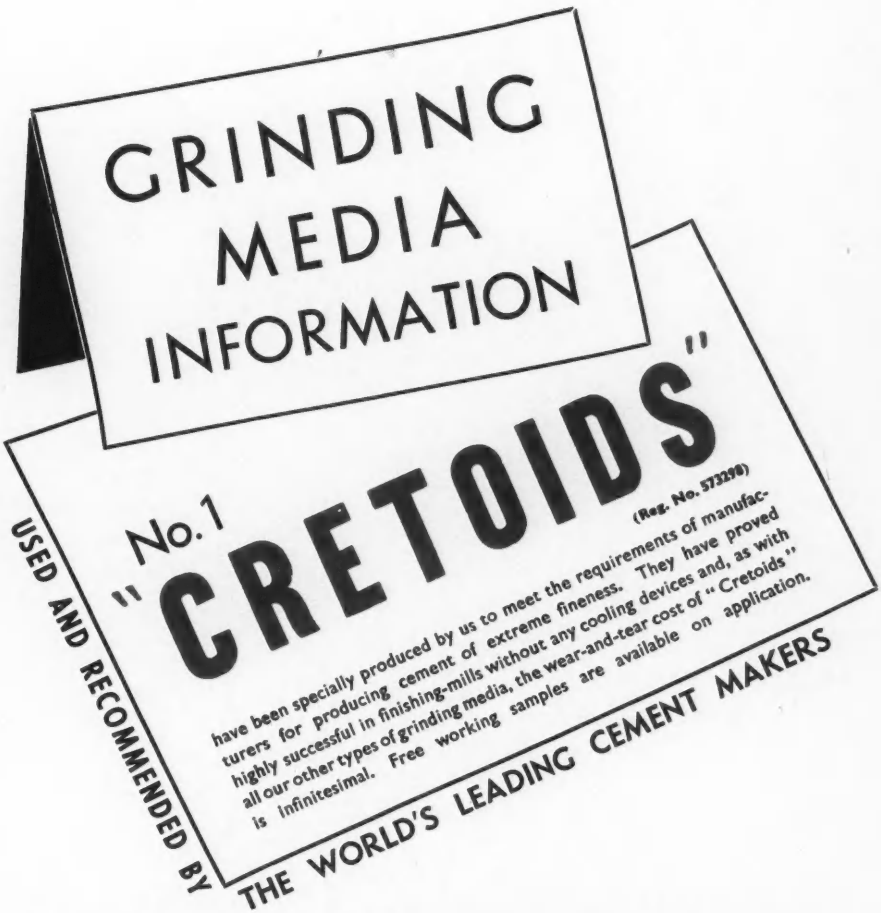
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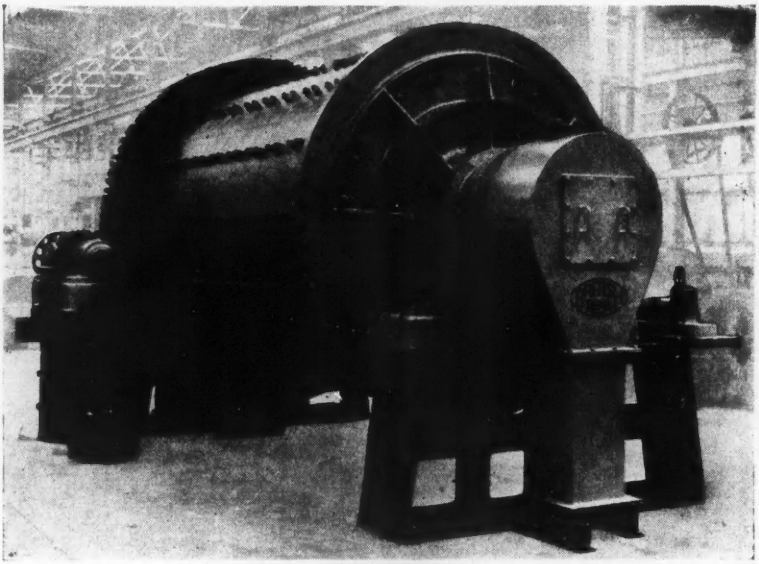

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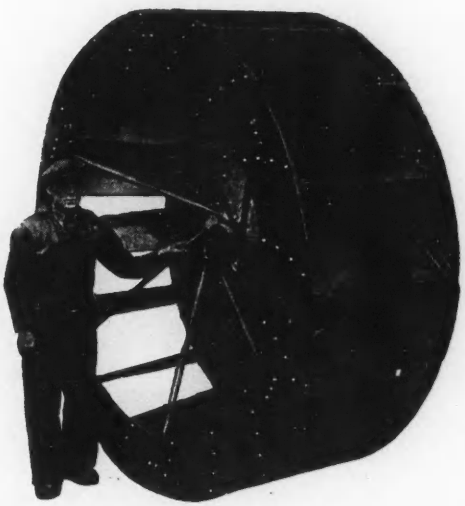

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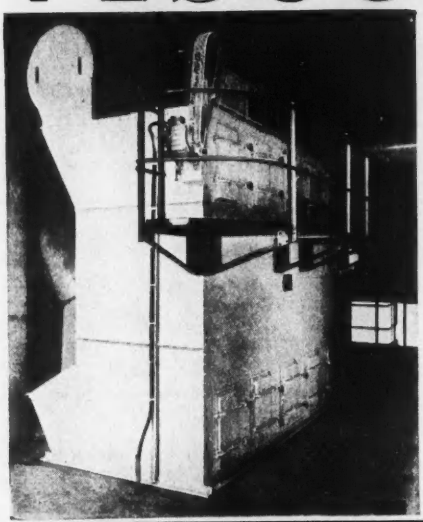

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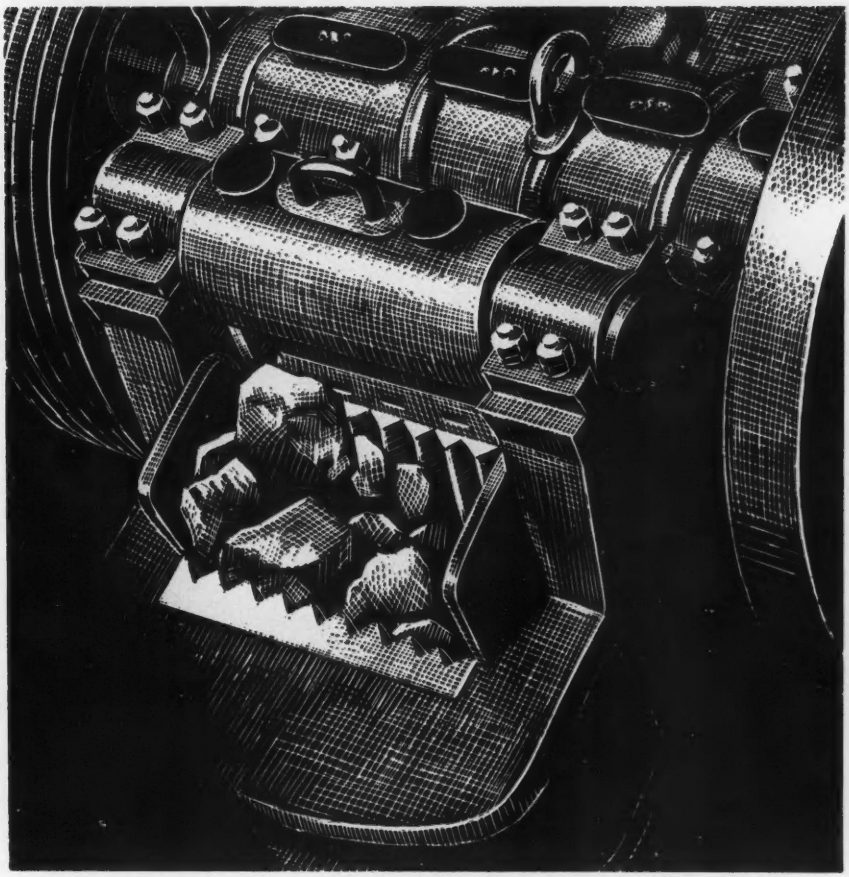

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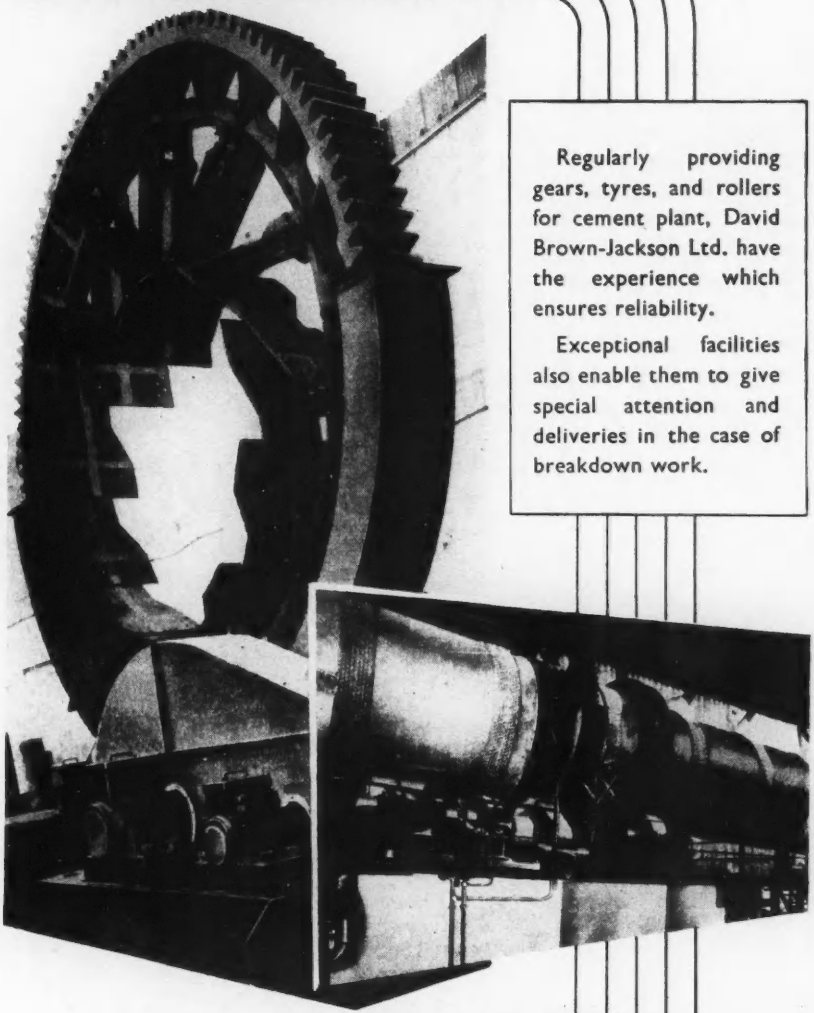

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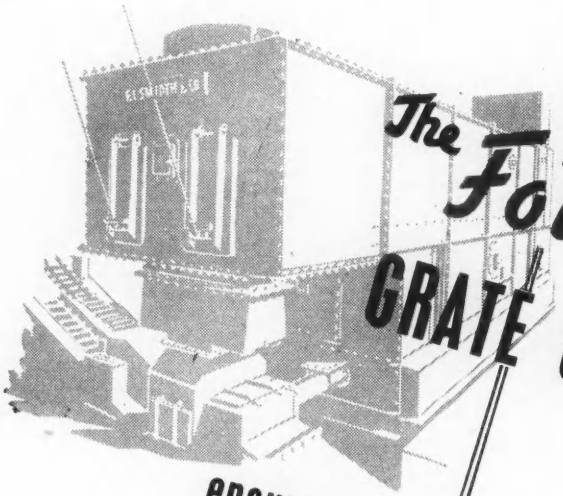

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VOLUME XXVI. NUMBER 1 JANUARY, 1953

Specification Tests for Heat of Hydration of Cement*

BY T. W. PARKER, Ph.D., F.R.I.C., AND R. W. NURSE, M.Sc., F.Inst.P.

SUMMARY.—The factors affecting the reproducibility of test results on the heat of hydration of cement are analysed and experimental data on the variability are given. The British Standard and U.S.A. Federal test procedures are compared, and it is suggested that either apparatus and procedure may be used in testing to the other specification. The requirement for results in triplicate improves the "confidence limits" in the B.S. procedure as compared with the U.S.A. procedure.

The Heat of Dissolution Method.

In the British Standard (B.S. 1370 : 1947)[1] for low-heat Portland cement a test is described for the measurement of heat of hydration by the differential heat of dissolution method. The procedure of this test will be discussed from the point of view of the accuracy of the final result.

The solvent consists of 388 c.c. of 2N nitric acid in which 9·6 c.c. of hydrofluoric acid (40 per cent. by weight of water) has been dissolved. The heat of hydration at any age is obtained by subtracting the heat of dissolution of a sample of cement hydrated for the given period from the heat of dissolution of the unhydrated cement.

The calorimeter for the determination of heats of dissolution is of simple construction. The body is a thermos flask of about 1 pint capacity, closed by a thick cork which supports a Beckmann thermometer reading to 0·001 deg. C. by interpolation, and a funnel through which the sample is introduced. Stirring is effected by means of a glass paddle operated by a constant-speed motor. *Fig.* 1

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shows the calorimeter. All parts which come into contact with the acid solution are protected by a wax coating.

The solution of the sample of cement takes about ten minutes, and it is important to reduce to a minimum the heat gained or lost through the walls of the calorimeter, the lid, the stirrer, and other parts during this period. For this reason a test is included in the specification for the selection of flasks of low heat-loss. Allowance must also be made for the heat generated by stirring; various types of stirrer were tested before selecting the ordinary paddle pattern.

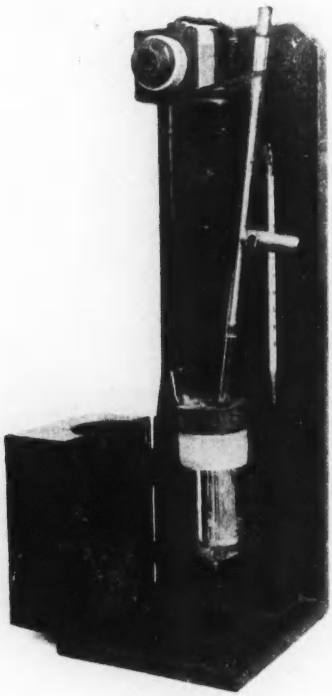

Fig. 1

For example, the centrifugal-type stirrer specified in U.S.A. Federal Specification No. SS-C-1586 was rejected because it generated 0·8 calorie per minute compared with 0·2 calorie per minute for the paddle type, while the effect on the rate of solution was negligible (*Fig.* 2).

In order to avoid the use of electrical calibration, the water equivalent of the calorimeter is determined from the known heat of dissolution of zinc oxide. The purity of this material is adequately controlled by specifying reagent quality,

but the rate of solution and the heat of dissolution also depend on its physical state. *Fig.* 3 shows the rate of solution of zinc oxide after various treatments, and *Fig.* 4 the gain or loss in weight when the ignited oxide is kept at various relative humidities. As a result of these tests the Specification requires the zinc oxide to be ignited for one hour at 900 deg. C. to 950 deg. C., cooled in a desiccator over anhydrous calcium chloride, and ground to pass a 60-mesh B.S. sieve. For each determination 7 gr. of this oxide are re-heated to 900 deg. C. to 950 deg. C. for five minutes and then cooled for from 2½ to 5 hours in the desiccator. Before this procedure was adopted the probable error of a single determination of the water equivalent was 1·25 cal. (in approximately 405 cal.); afterwards it fell to 0·32 cal.

Some slight increase in accuracy might have been obtained by weighing the contents of the calorimeter instead of measuring by volume; the final overall

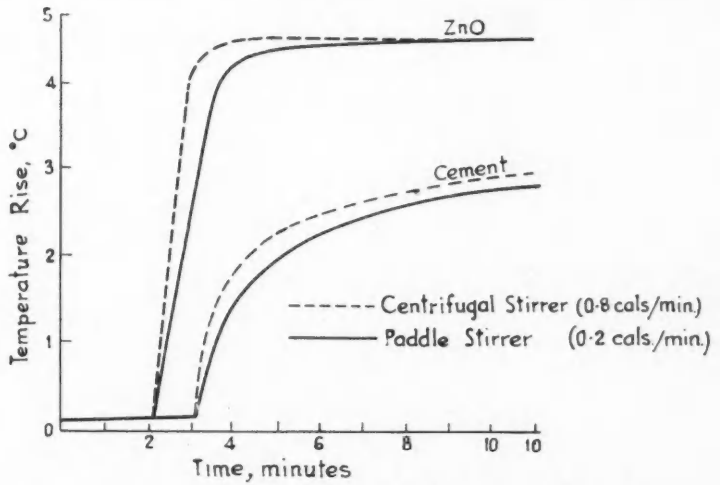

Fig. 2.—Effect of Type of Stirrer on Rate of Solution of ZnO and Cement.

errors reported later show, however, that sufficient accuracy is achieved without this complication. The procedures for the determination of the heats of dissolution of unhydrated and hydrated cement are fully described in the specification.

Preparation and Treatment of Samples.

It was found unnecessary to specify mechanical mixing of the cement paste for the hydrated sample, nor did the experience of the mixer have any effect on the variability of samples. A typical series gave the following results for six determinations of the heat of dissolution in calories per gramme at seven days: experienced operator, mean 526·3—standard deviation 1·6; inexperienced operator, mean 527·7—standard deviation 1·6. A short series of tests was

carried out in which the temperature of the mixing water and cement was varied. The results do not justify calculation of standard deviations, but *Table* 1 shows that the mixing temperature need not be closely specified.

TABLE 1.—HEAT OF DISSOLUTION AT 7 DAYS : PORTLAND CEMENT

Temperature of mixing	Heat of dissolution (calories per gramme)
32 deg. Fahr.	529·8 ; 528·9 ; 528·4
64 ,,	529·5 ; 531·2 ; 530·9
67 ,,	530·0 ; 530·3 ; 530·1

The effect of storage conditions was investigated for one low-heat Portland and one Portland cement ; two operators took part, each making three independent tests on each cement and for each storage condition. The storage conditions

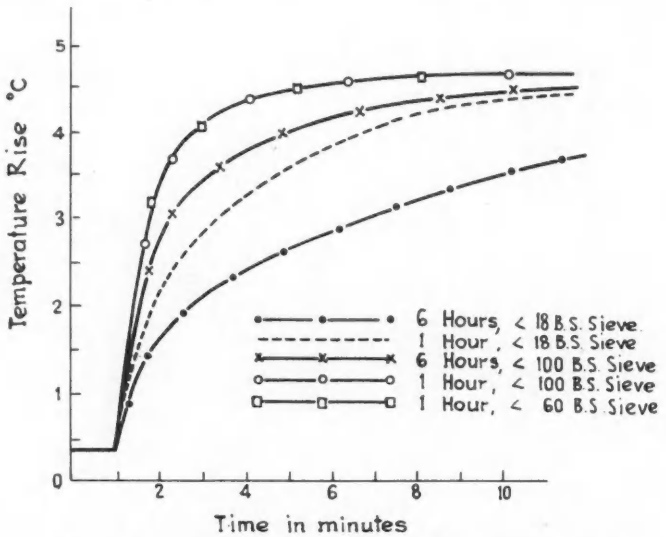

Fig. 3.—Effect of Heat Treatment at 900-950 deg. C. and Fineness on Rate of Solution of Zinc Oxide.

tested were : (1) 70 deg. Fahr. for seven days ; (2) 64 deg. Fahr. for one day, 70 deg. Fahr. for six days ; (3) The vial containing the cement was rotated at 90 revolutions per minute for one day at 64 deg. Fahr., followed by 70 deg. Fahr. for six days ; (4) As (1) but for 28 days ; (5) As (2) but 70 deg. Fahr. for 27 days ; (6) As (3) but 70 deg. Fahr. for 27 days. The results are given in *Table* 2. Since the cement in a mass of concrete is subjected during its early hydration period to a rise in temperature, rapid at first and gradually declining, a mixed storage

condition such as (2) has frequently been suggested. Since the results obtained under conditions (1), (2), and (3) or (4), (5), and (6) were not significantly different it is preferable to choose the simplest conditions, namely (1) and (4).

TABLE 2.—MEAN VALUES AND STANDARD DEVIATIONS FOR VARIOUS STORAGE CONDITIONS

Storage Conditions	Heat of Dissolution		
	Mean value. Low-heat cement (cal. per gramme)	Mean value. Normal Portland cement (cal. per gramme)	Overall standard deviation (percentage of mean)
(1)	542·8	530·8	0·22
(2)	541·8	531·0	0·34
(3)	542·3	531·9	0·25
(4)	525·4	513·9	0·20
(5)	526·4	514·6	0·21
(6)	527·2	514·6	0·31

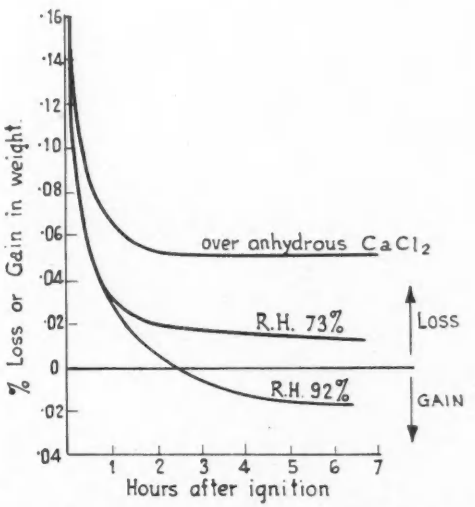

Fig. 4.—Change in Weight of ZnO ignited at 900 deg. C. (zero-line represents weight before ignition).

Reproducibility of Results.

Standard deviations were calculated for the results of two observers using the specified procedure, each standard deviation being based on six results from each observer. The values are given in *Table* 3. For comparison, results obtained using the U.S.A. Federal Specification[2] method are quoted[3].

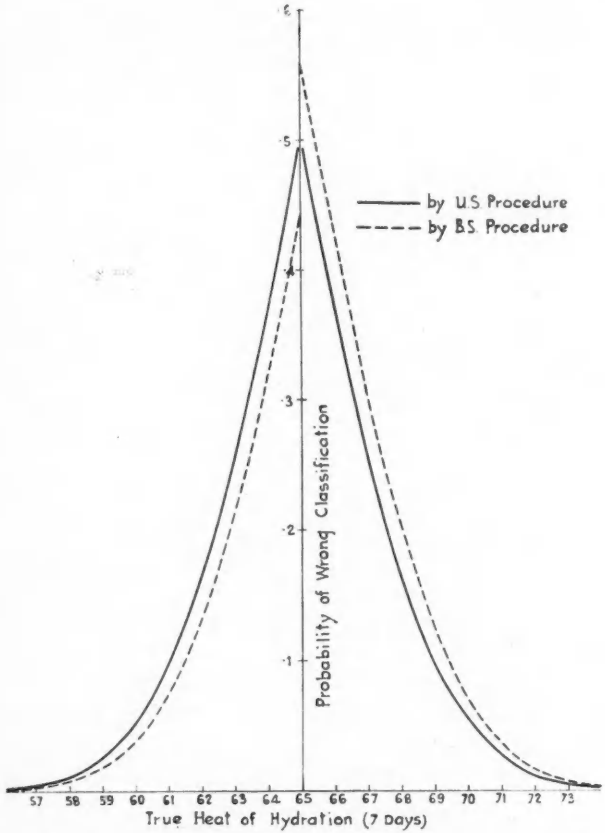

Fig. 5.—Heat of Hydration of Cement. Single determination.

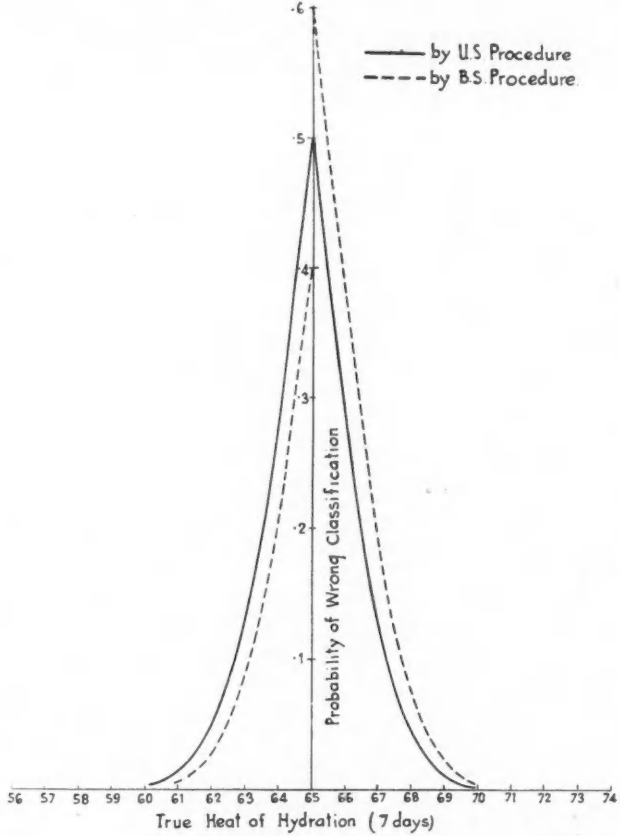

Fig. 6.—Heat of Hydration of Cement. Triplicate Determinations.

TABLE 3.—STANDARD DEVIATIONS

Type of Test	Standard Deviation (cal. per gr.)	
	B.S. No. 1370 procedure	U.S.A. SS-C-1586 procedure[3]
Water equivalent	0·32	1·1
Heats of dissolution :		
(a) Unhydrated cement	0·60	1·5
(b) 7 days' hydrated cement	1·40	2·7
(c) 28 days' hydrated cement	1·20	1·4

Three determinations each are specified in the British Standard for the heats of dissolution of unhydrated cement, and for cement hydrated at seven and 28 days. The standard deviation of the means of these three and the calculated standard deviations of the final heats of hydration are given in *Table* 4.

TABLE 4

Heats of Dissolution (cals. per gr.). Standard deviation of means		Heats of Hydration (cals. per gr.). Standard deviation of differences
7 days' hydrated	0·80	0·9. Heat of hydration at 7 days
Unhydrated	0·35	
28 days' hydrated ..	0·70	0·8. Heat of hydration at 28 days

Comparisons of B.S. and U.S.A. Federal Procedures.

The results given in *Table* 3 indicate a slightly better reproducibility when the B.S. procedure is used. When the chance of wrong classification is calculated for single determinations, however, there is nothing to choose between the two methods. *Fig.* 5 shows this calculation for the 65 cal./gr. classification of B.S. No. 1397 at seven days. A discontinuity is introduced into the curve for the B.S. procedure because of the requirement that results be to the nearest calorie, otherwise there would be an equal chance of right or wrong classification at 65 cal. per gr. If a 5 per cent. chance of wrong classification is considered to be reasonable, then by the B.S. procedure this occurs at 60 and 71 cal. per gramme and by the U.S.A. procedure at 60 and 70 cal. per gramme.

A significant increase in reliability is obtained, however, by the requirement for triplicate determinations in the B.S. procedure. A probability curve for triplicate determinations is given in *Fig.* 6. Comparing the values for a 5 per cent. probability of wrong classification on the basis of single determinations by the U.S.A. procedure and triplicate determinations by the B.S. procedure, the following values of the heat of hydration (in calories per gramme) are obtained : U.S.A., 60 and 70 ; B.S., 63 and 68. Apart from the question of reproducibility, the B.S. and U.S.A. Federal procedures are so similar that it may be assumed with reasonable confidence that results obtained by either are acceptable under the terms of the other specification.

ACKNOWLEDGEMENTS.—The authors wish to thank Miss S. Wickham-Jones and Mr. A. C. Riddle who assisted with the experimental work. The paper is published by permission of the Director of Building Research.

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[1] British Standard No. 1370 : 1947. British Standards Institution, London.
[2] Federal Stock Catalogue, Section IV, part 5. U.S. Printing Office, Washington.
[3] E. S. Newman. J.Res. N.B.S. 1950, 45 (5), 411.

Methods of Determining the Rate of Settling of Hydrated Lime Slurries.

IN the September, 1952, number of the Bulletin of the American Society for Testing Materials, from which the following is abstracted, Mr. R. K. Thomas compares two methods of determining the settling rate of hydrated lime slurries. The differences between the methods are essentially in the manner in which the original slurry is made up. The first, or "Proposed A.S.T.M. Method", requires mixing in a large volume of water in the tube actually used for comparison; the second, or "Modified Method," requires that the sample be first made into a thick putty, using a small amount of water, and allowed to stand in putty form before being diluted to the proper volume for the test. This second method was proposed as it was thought to give a more intimate mixture of the lime with the water than would be possible using only a shaking procedure.

Proposed A.S.T.M. Method.

Place 10 g. of lime hydrate in a 100-ml. graduated flask (internal diameter about 2·4 cm.), wet with about 50 ml. of distilled water at 70 deg. F., and shake vigorously to complete suspension. Allow to stand at 70 deg. F. for two hours and then make up to the 100-ml. mark with distilled water at 70 deg. F. Shake the flask and contents vigorously and then allow to stand undisturbed for 24 hours. Record the sedimentation height of the final volume in millilitres after standing for ½, 1, 2, 4, and 24 hours.

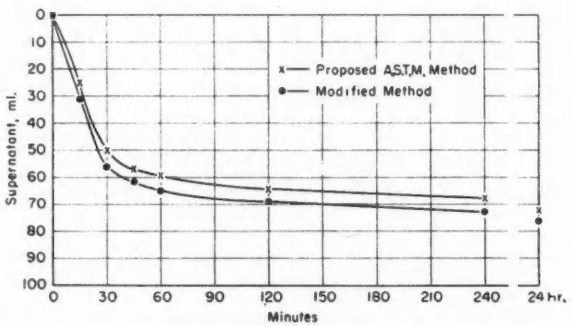


Fig. 1.—Average Settling Rates of Sample No. 1.

Modified Method.

Weigh accurately 10 g. of the dry hydrated lime into a dry 150-ml. beaker and mix with sufficient distilled water (70 deg. F.) to make a thin putty (15 to 20 ml.). The lime and water should be stirred thoroughly with a rubber-tipped glass stirring-rod during the mixing process. Allow the putty to stand covered for half an hour at a temperature of 70 deg. F. Wash the contents of the beaker into a dry 100-ml. graduated cylinder with a glass stopper and scrub the beaker thoroughly to remove all lime adhering to the sides. Dilute to the 100-ml. mark, shake vigorously for one to two minutes and allow to stand undisturbed at a temperature of 70 deg. F. Record the level of the sediment at 15-minute intervals for one hour and then at the 2–, 4–, and 24-hour periods.

The programme was limited to a consideration of settling rates of hydrated limes, and samples were selected to give a range of settling characteristics. Samples Nos. 1 and 2 are dolomitic hydrated limes, both of which are fast-settling. Sample No. 3 is a high-calcium hydrate having a slow rate of settling. Tests were made in eight laboratories, and the averages of the results for sample No. 1 are shown in Fig. 1. The results obtained using the proposed A.S.T.M. method

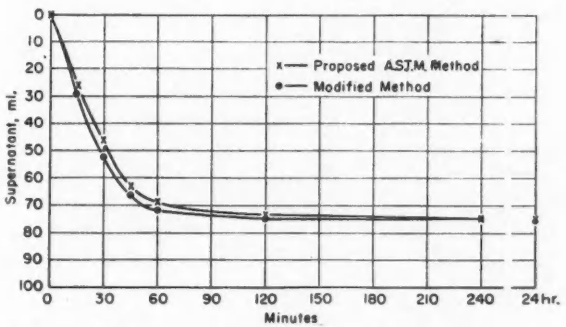


Fig. 2.—Average Settling Rates of Sample No. 2.

are variable, particularly up to 45 minutes, but after one hour all are fairly consistent. Results obtained by the Modified Method likewise show considerable variation although the results obtained after one hour of settling are again consistent.

The average of the results obtained using sample No. 2 are shown in Fig. 2, and for the proposed A.S.T.M. method results obtained in the several laboratories vary in the same way as those for sample No. 1. The Modified Method indicates consistent results in all but two cases, and again the divergence which can be noted at the earlier ages becomes smaller after the sample has been allowed to stand for one hour.

The average of the results obtained on sample No. 3 are shown in Fig. 3. Those obtained by the proposed A.S.T.M. method are consistent, with one exception. The results of the Modified Method by seven laboratories reporting

on this method show four good checks at one time and three good checks at another time, the two sets being rather out of line with each other.

The average values of the three samples indicate the same rate of settling, regardless of the method of testing adopted. The marked difference is that the results by the Modified Method show a slightly faster rate of settling. The similarity of the rates is shown by the curves. An interesting fact also shown by the curves is the better check of average results with a slower-settling lime.

Comparison of the values at the different time intervals shows a comparatively wide diversity of results obtained in the several laboratories regardless of the method used. Neither method shows any marked superiority from the standpoint of reproducibility of results in a large number of laboratories working on similar samples.

On the basis of the extreme range experienced at each time interval, the proposed A.S.T.M. method appears to give closer co-ordination. The greatest differences in results are at 15 and 45 minutes. These variations can be partly discounted since more laboratories reported results at these time intervals using

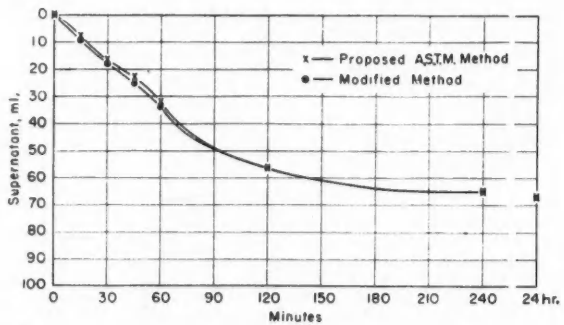


Fig. 3. Average Settling Rates of Sample No. 3.

the Modified Method than was the case using the proposed A.S.T.M. method. With the proposed A.S.T.M. method results were reported by only three laboratories on samples Nos. 1 and 2 and by only two laboratories on sample No. 3, whereas the results by the Modified Method were reported by seven laboratories for samples Nos. 1 and 2 and by six laboratories for sample No. 3. Thus the chance of extreme variations was considerably lessened.

Results by the Modified Method appear to be more consistent at all the time intervals. The results of the tests in any one laboratory on any one sample are similar throughout the whole of the test in more instances with this method. Both methods gave similar results for the rates at and after settling for one hour, and final volumes were generally the same, both as regards the same method and the two methods. The differences in results between the two methods were less for a slow-settling time than for a fast-settling time, taking into consideration all the time intervals.

The influence of the size of the flask on the results was also studied. Results were reported in terms of millimetres of height as well as millilitres of volume. All tubes used for the test were standard 100-ml. flasks and there was no marked difference in the height or distance of fall, and the comparative height of the tube used appears to be of little consequence ; this would, of course, be true only where such differences in size are slight and would preclude the use of equipment having extremely wide variations in size.

Small variations in the temperature at which the test is made do not appear to influence greatly the results reported by different laboratories, but the temperature has an effect on the results of tests made in the same laboratory under the same general conditions.

Conclusions.

The many factors entering into the determination of a rate of settling of hydrated lime slurry appear to make very close reproducibility of results in different laboratories nearly impossible. The results depend to too great an extent on the skill of the person making the test. Either method studied distinguishes only between a fast- and slow-settling hydrate. Checks of rates to limits of, say, 1 to 2 ml., are not possible.

The Modified Method, although requiring more manipulation on the part of the tester, is slightly better and should give results which could be checked within reasonable limits in another laboratory.

New Cement Works in Uganda.

THE inauguration of a cement industry in Uganda is largely due to the demand for cement for the construction of the Owen Falls dam for the Uganda Electricity Board. The Board, whose chairman is Mr. C. R. Westlake, M.I.E.E., was entrusted by the Government with the responsibility for the establishment of the cement works at Tororo, described in the May, 1952, number of this journal. The design and supervision of the erection of the buildings and civil engineering works were by the Public Works Department under the direction of Mr. S. S. Tindall, C.B.E., and Messrs. Kennedy & Donkin (E. A.) designed and supervised the electrical installation. The geological investigations were carried out by Dr. K. A. Davies, C.M.G., O.B.E., and the Uganda Geological Survey, and investigations in connection with the raw materials were by Mr. R. W. Nurse of the Building Research Station and Dr. A. J. V. Underwood. Messrs. Stirling-Astaldi (E. A.), Ltd., were the main contractors.

New Cement Works for Pakistan.

A CEMENT factory costing five million dollars is to be built in Punjab in northern Pakistan, as part of Canada's share of the Colombo plan for developing countries in south and south-east Asia. The factory is to be built by a new company formed by five Canadian firms. The factory will produce more than 100,000 tons of cement a year for an irrigation and reclamation project.

Relation Between Specific Surface and Other Properties of Cement.

THE amount of work required to test cement by ordinary methods of determining the chemical composition, fineness, setting time, soundness, etc., and the delay in obtaining the results because of the time required to elapse before making some of the tests, makes any less laborious and more rapid method worthy of consideration. In recent numbers of "Revue des Materiaux de Construction," Dr. M. J. M. Jaspers describes a method of determining the quality of cement based on the relation between the fineness and the other properties. A summary of the article is given in the following.

The properties are divided into two groups, namely the independent variable properties, that is the mineralogical composition and the specific surface, and the properties that are functions of the independent variable properties, that is the rate of setting and hardening, the volumetric stability, the heat of hydration, the workability, and the resistance to chemical attack. If graphs are drawn to relate these properties to the two independent variables, only the latter need be determined experimentally when testing a cement.

Although the principle described is simple, the application is complex, but it is possible to obtain a practical solution which is applicable to very many cases if some simplifications are made. It is first necessary to determine which properties are dependent on the specific surface, which on the composition, and which on both the specific surface and composition. To simplify the investigation it is first assumed that the mineralogical composition is constant, leaving the specific surface the only independent variable.

Determination of Specific Surface.

The values of the specific surface determined by a turbidimeter and by a permeability apparatus differ, the value obtained by the Blaine permeability apparatus being greater than that obtained by the Wagner turbidimeter. It is thought that the results obtained by the Blaine apparatus are more correct. A third method[1] is that upon which the investigations made by Dr. Jaspers are based, and is the application of the granulometric equation

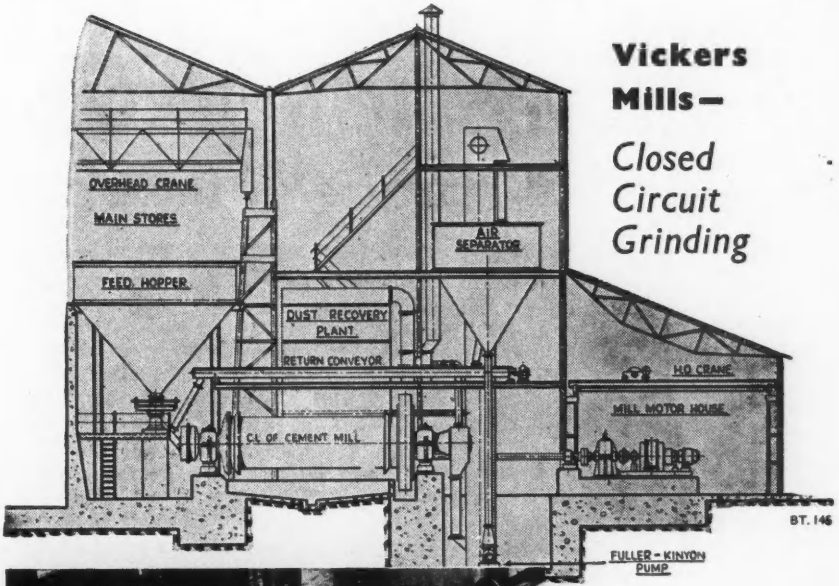
$$R = 100e^{-bx^n} \quad .. \quad .. \quad .. \quad .. \quad (1)$$

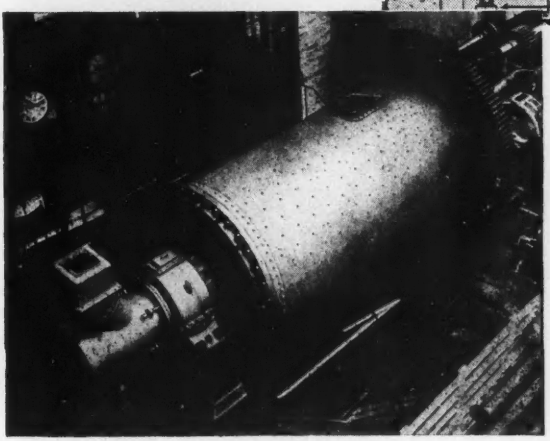
in which R is the percentage retained on a sieve the size of the mesh of which is x; b is a constant and e the base of natural logarithms; n is the tangent of the angle between the granulometric curve and the abscissa; for cement it is about unity, although more accurate values were used in the investigations. The second logarithm of equation (1) gives

$$\log\left(\log \frac{100}{R}\right) = C + n \log x \quad .. \quad .. \quad .. \quad .. \quad (2)$$

A graph with co-ordinates $\log\left(\log \frac{100}{R}\right)$ and $\log x$ can be drawn and from this the complete granulometric composition of a cement can be obtained by measuring

Vickers Mills— *Closed Circuit Grinding*





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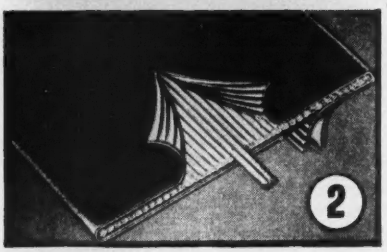

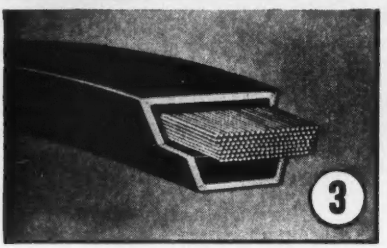

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the percentage retained on one sieve only. Dr. Jaspers states that tests made by him have established the accuracy of the method. The specific surface S_A is determined from the following equation[2].

$$S_A = \frac{36{\cdot}8 \times 10^4 \times 1{\cdot}75}{\mu_a n w} \text{ sq. cm. per gramme}$$

in which μ_a is the abscissa of the point of intersection of the straight line, representing the granulometric composition on the graph having co-ordinates $\log\left(\log \frac{100}{R}\right)$ and $\log x$, with the ordinate $R = 36{\cdot}8$ per cent. ; n is the inclination (tangent) of the straight line ; w is the specific gravity of the cement. Tests show that the relation between S_A and the specific surface S_T determined by a turbidimeter is linear, and is given by

$$S_T = 0{\cdot}19\, S_A + 448$$

from which, knowing S_T, the corresponding value of S_A, on which the relations in the following are based, can be determined.

Mechanical Resistance.

The strength R_m, or mechanical resistance, of set cement depends on the reactions between the water and the mineralogical constituents of the cement. The reactions occur on the surface of the solid phase and consequently the quantity Q_t of cement hydrated is a mathematical function of the specific surface S. Likewise R_m is a function of S. It can be established that Q_t is a linear logarithmic function of S of the form, $\log Q_t = aS - b$, and graphs based on experimental results can be drawn to relate Q_t to R_m. Consequently a relation between R_m and S can be established.

It is, however, possible to obtain this relation directly. From the results of tests it is determined that the mechanical resistance, that is the tensile strength R_T or compressive strength R_C is a linear function of the specific surface. The strength of at least two samples of cement of different finenesses (specific surface) are determined and a graph showing the relation of R_T or R_C to S_A is drawn. The graph can then be used to determine R_T or R_C for any sample of cement having the same mineralogical composition if the specific surface S_A of the cement is known. *Figs.* 1 and 2 are typical graphs for R_C and R_T respectively at various ages and are based on the tests made to establish the relationship of R_m to S. The first point on each curve is for samples of cement taken from the grinding mill at a cement works. The second to fifth points are for the same cement after it has been ground in a laboratory mill to various degrees of fineness.

Setting Times.

Tests also showed that the times from adding the water to the cement to the beginning and end of the setting period are also linear functions of the specific surface as is seen in *Fig.* 3, in which the time in minutes is plotted against the specific surface S_A (calculated by the method described previously) and against the specific surface S_T determined by a turbidimeter. The amount of water in the samples upon which *Fig.* 3 is based was 27·5 per cent. The mineralogical composition of all the samples was the same.

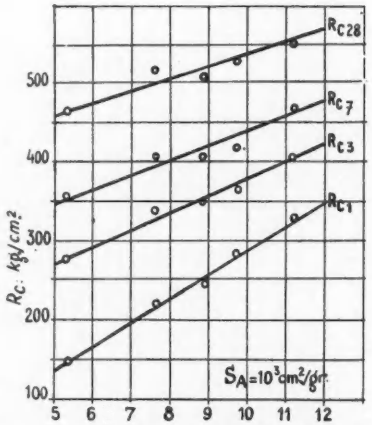

Fig. 1.—Compressive Strength as a function of Specific Surface.

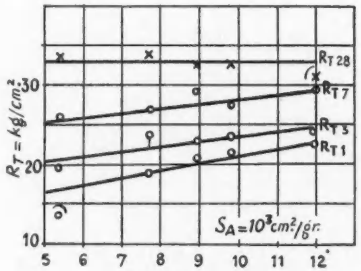

Fig. 2.—Tensile Strength as a function of Specific Surface.

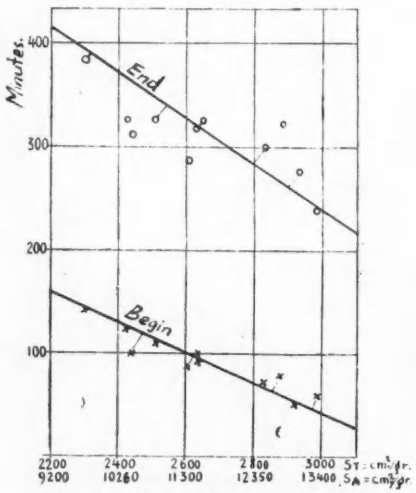

Fig. 3.—Setting Time as a Function of Specific Surface.

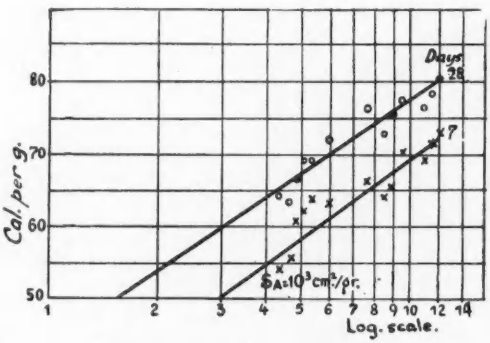

Fig. 4.—Heat of Hydration as a Function of Specific Surface.

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Heat of Hydration.

The heat C_H generated during setting appears to be a linear function of the logarithm of the specific surface, that is

$$C_H = a \log S + b$$

The heat of hydration (calories per gramme) was determined by the method described in the Federal Specification of the United States of America, and the results of some of the tests on samples of cement having the same mineralogical

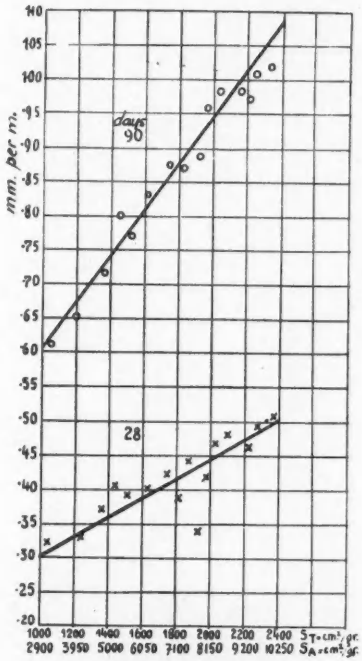

Fig. 5.—Shrinkage as a Function of Specific Surface.

composition are given in *Fig.* 4. The samples in this case were of the same cement as used in the tests for mechanical resistance, the results of which are given in *Figs.* 1 and 2.

Other Properties.

The results of tests given in *Fig.* 5 show that shrinkage is also a linear function of the specific surface. The shrinkage was measured by a Huggenberger apparatus on prisms, 40 cm. long and 7 cm. square, of 1 : 3 mortar.

The Vicat or Gillmore test to determine the proportion of water necessary to produce a paste of normal consistency can be used to measure the workability of a cement. It has been established[3] that the proportion of water is a linear function of the specific surface.

So far the author had not made tests to establish the relation between the specific surface and the expansion, liability to segregation, and resistance to chemical attack, nor has much work been done on establishing the relation between the properties of cement and the two independent variables, namely specific surface and mineralogical composition. The fact that the tests described relate to cements of constant mineralogical composition does not detract from the value of the results because this condition is generally the case in works where the composition of the slurry and the quality of the fuel are controlled.

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[1] P. Rosin, E. Rammler, and K. Sperling. Bericktsfolge des Reichskohlenrates, 25, C52 (Berlin : 1933). Beiheft Verfahrenstechnik, 1937, p. 161 ; 1940, p. 150.

[2] W. Anselm. Zerkleinerungstechnik und Staub. (Dusseldorf : Deutscher Ingenieur-Verlag).

[3] J. A. Swenson, L. A. Wagner, and G. L. Pigman. Effect of granulometric composition of cement on the properties of pastes, mortars, and concretes. National Bureau of Standards Research Paper 777 (1935).

Delivery of Loose Cement in Sweden.

IT is stated that in the larger cities of Sweden about half of the cement deliveries are made in containers such as that shown in *Fig.* 1. The lorries carry two or three vertical pressure tanks with a total capacity of from 5 to 14 tons, which can be increased to 28 tons by the addition of a trailer. From these tanks the cement is discharged into silos at a rate of up to one ton per minute. The unloading machinery has no moving parts except the compressor.

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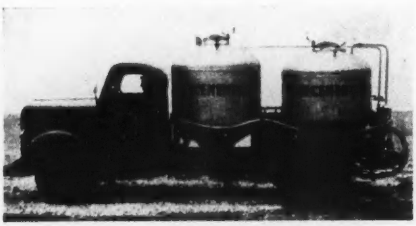

Fig. 1.

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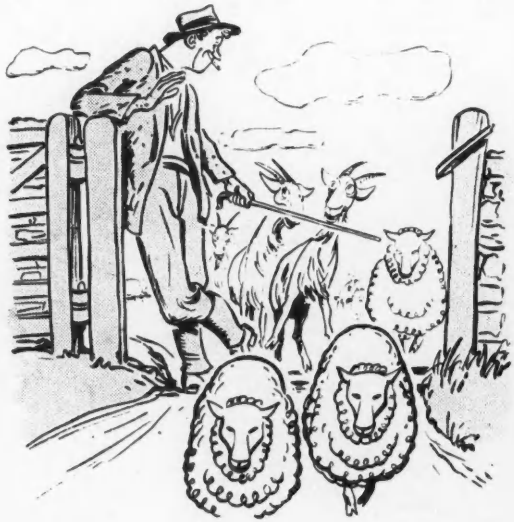

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